

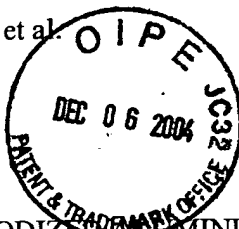
**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

IN RE APPLICATION OF: Yixing Lin et al.

SERIAL NO.: 10/071,869

FILED: February 8, 2002

FOR: HALOGEN-RESISTANT, ANODIZED ALUMINUM  
FOR USE IN SEMICONDUCTOR PROCESSING  
APPARATUS



§ GROUP ART UNIT: 1742

§ EXAMINER: J. A. Combs-Morillo

§ Attorney Docket No.: AM-6846

Date: November 10, 2004

**DECLARATION OF INVENTORS**  
**UNDER 37 CFR § 1.132**

Hon. Commissioner for Patents  
P.O. Box 1450  
Alexandria, Virginia 22313-1450

Sir:

I hereby declare that I, Hong Wang, am an inventor of the invention claimed in the subject patent application. This Declaration is being filed in response to the final Office Action mailed October 6, 2004, and in response to suggestions by Group Art Unit 1742 SPE Roy King in a telephone interview with applicants' attorney on November 5, 2004 regarding a CIP application of the present application.

**CERTIFICATE OF MAILING UNDER 37 CFR § 1.10**

I hereby certify that this paper and any additional correspondence referred to as included with this paper are being deposited with the U.S. Postal Service on the date shown below with sufficient postage as U.S. EXPRESS MAIL NO. ED 273473571 US in an envelope addressed to: Mail Stop AF, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Date: December 6, 2004

Shirley L. Church  
Shirley L. Church, Reg. No. 31,858

I, Hong Wang, further declare and state as follows:

1. This Declaration is being prepared by me and reviewed and signed by other inventors who are available within a reasonable time period (to enable a timely response), to indicate their agreement with the contents of this Declaration.
2. I received a Ph.D. in physics from Florida Atlantic University in 1993. Since that time, I have published more than 30 journal and conference papers. In addition, I have contributed as an inventor to more than 20 patent applications, several of which have already issued as patents.
3. My work has spanned more than 20 years in the field of semiconductor processing, device yield enhancement and failure analysis, and material science. This includes more than 4 years as a Senior Engineering Manager in Materials Science and Process Engineering at Applied Materials, Inc., Santa Clara, California, and more than 4 years as Process Integration Manager in a silicon wafer fab, where more than 30,000 wafers per month are processed.
4. This Declaration is in response to the final Office Action mailed October 6, 2004, in which Claims 8 - 20, 24, 25, and 28 - 34 are finally rejected. In that Office Action, the principal reference cited alone and in combination with other references as the basis for rejection of our claims is the Miyashita et al. reference, U.S. Patent No. 5,039,388. This reference is said to anticipate under 35 USC § 102(b) or to make obvious under 35 USC § 103(a), our claimed subject matter. We contend that the Miyashita et al reference teaches an invention which solves a different problem than the problem which is solved by our invention, and that the teachings of the Miyashita et al. reference are distinct from the teachings of our invention.

5. The Examiner has argued that the Miyashita et al. reference mentions a maximum particle diameter which falls within the particle distribution range of the high purity aluminum alloy recited as part of our claimed method. However, we contend that with respect to the physical behavior which is observed for the aluminum alloy, a maximum particle diameter is not indicative of a particle size distribution and is distinctly different in terms of what it teaches. In addition, the Examiner has argued that the Miyashita et al. reference teaches use of an anodic oxidation layer over an aluminum alloy to increase corrosion resistance. However, the corrosion resistance the Miyashita et al. reference is addressing is the corrosion/erosion resistance of the anodized layer itself, and not the corrosion resistance of the aluminum alloy from which the anodized layer is grown. Our invention relates to a method of producing an anodized layer where the substrate used as a starting material subsequently prevents or reduces corrosion of the underlying substrate. The Examiner cites Col. 2, lines 43 - 44 and 63 - 64 and Col. 3, line 9. This subject matter including adjacent text, i.e. Col. 2, lines 43 - 48 and lines 60 - 66, and Col. 3, lines 7 - 18 reads as follows: "However, aluminum electrodes having a surface layer formed by anodic oxidation in a bath containing sulfuric acid which are widely used in apparatus for high frequency plasma treatment suffer a heavy reduction in layer thickness due to corrosion during the treatment." . . . "Our further studies have taught us that an anodized layer formed by employing a chromate bath is barely corroded by active fluorine, but is durable for a long period of time without having any substantial reduction in thickness, as opposed to the conventional layer formed by employing a sulfate bath" . . . "The anodized layer formed in a chromate bath not only contributes to the production of a stable plasma, but also is highly resistant to corrosion by active fluorine gas. Such corrosion leads to a reduction in thickness during formation of a film of silicon dioxide or nitride during a wafer . . . . As it does not undergo a substantial reduction in its thickness, the (anodized) layer contains few particles that would contaminate a silicon dioxide or nitride film, or hinder its satisfactory etching." (Col. 3, lines 7 - 18.) It is clear that the concern in the Miyashita et al. reference is with changes in the thickness of the anodized layer and

particulates which may be generated from the anodized layer corrosion/erosion. This reference does not address the problem which our invention addresses, which is corrosion of the underlying aluminum substrate.

6. Our invention relates to a high purity aluminum alloy with a controlled particle size distribution and a controlled chemical composition where particular impurities present in the alloy are not permitted to exceed a maximum nominal amount. One who works in the art of metallurgy will recognize that there is a significant functional difference between particle size distribution and a maximum particle size; and, that there is a difference between an overlapping range of an element in a composition and the maximum amount of an element which may be present. These differences in the aluminum alloy become important when the concern is with formation of an anodized layer which is not porous to reactive species which corrode an underlying aluminum substrate.

7. Our invention relates to a method of forming an anodized layer over a high purity aluminum alloy, where the method of formation extends the performance lifetime of a semiconductor processing component formed from the anodized alloy, when the component is exposed to a halogen-containing environment. By controlling the initial particle size distribution and the composition of the high purity aluminum alloy, we have been able to reduce the formation of agglomerations of particles within the anodized layer as it grows and at the interface between the anodized protective layer and the underlying aluminum alloy. By controlling the initial particle size distribution in and the composition of the high purity aluminum alloy, so that a proper anodized protective layer is formed over the high purity aluminum alloy, we have extended the useful lifetime of a semiconductor apparatus component exposed to the harsh halogen-containing plasmas of the kind used during cleaning of the processing apparatus, for example. The formation of particulate agglomerations at the interface or within the anodized protective layer serves as a stress point which causes cracking and

failure of the aluminum oxide protective coating. In addition, since an anodized protective layer grows from the surface of the aluminum alloy, any impurity inclusion located near the alloy surface will remain at the interface between the growing anodized layer and the aluminum alloy or will move forward into the anodized layer. Such impurity inclusions are not chemically resistant and are easily attacked by contact with a halogen plasma, creating "tunnels" which lead to the underlying aluminum substrate. Particles generated at the aluminum substrate surface may exit through such tunnels in the anodized layer and generate particulates on semiconductor wafers which are being processed using the processing chamber or component.

8. In addition to the initial particle size distribution, the composition of the aluminum alloy, notably the maximum concentration of mobile impurities which are present in the alloy, plays a very important role in many semiconductor processing apparatus applications. There migrating impurities interfere with the formation of the protective anodized layer and may migrate during the lifetime of the semiconductor processing apparatus in a manner which is harmful to semiconductor wafers which are being processed in the apparatus. The migrating impurities which migrate during formation of the anodized layer produce defects in the anodized layer which subsequently cause cracking and tunnel formation which permits halogen species to penetrate to the underlying aluminum alloy substrate. As a result, the underlying aluminum surface becomes corroded, generates particulates, and these particulates exit through the defective anodized layer to deposit on semiconductor device surfaces which are being processed in the apparatus. If the cracking of the anodized layer is severe enough, sections of the anodized layer may flake off, leading to further corrosion and contamination.

9. To avoid the problem described above, we developed a specialized high purity aluminum alloy with controlled initial particulate size distribution and with particular maximum limits on the concentration of mobile impurities of the kind which can form

agglomerates during fabrication of the anodized layer and during use of an article or component fabricated from the aluminum alloy.

10. The Examiner has argued that since the Miyashita et al. reference describes a maximum initial particle size which is smaller than the maximum initial particle size specified in our particle size distribution, this anticipates or makes obvious our invention. We are concerned with the amount of particles present over a given range of particle sizes, not just a maximum particle size. If there are a sufficient number of particles present at a particle size which permits the formation of harmful agglomerations during fabrication and use of the anodized aluminum article, this will cause the overlying protective anodized layer to fail.

11. We have carried out substantial experimentation with respect to the performance value of the combination of an anodized layer created over the surface of our specialized aluminum alloy. The initial experimental results were presented in our patent application as originally filed in Paragraphs 56 and 57.

12. Subsequent to filing of the present application, we have sold more than 80 process chamber liner packages which make use of our invention to protect very expensive large semiconductor processing chambers. These liners have our high purity aluminum alloy as the base structure, with an anodized layer protecting the surface of the high purity aluminum alloy. These liners are used to protect process chamber which cost about \$450,000 to \$500,000 each. In December of 2002, Applied Materials, Inc. won the prestigious Editor's Choice Best Product Award from Semiconductor International magazine for its Dielectric Etch eMax™ Centura® system, which makes use of our anodized layer-protected specialized high purity aluminum alloy.

13. We have continued to develop data which illustrates the advantages of our inventive high purity aluminum alloy, which is often used in combination with an anodized layer to

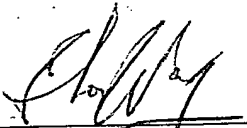
provide exceptional performance results. Attached to this Declaration are two customer support pamphlets which illustrate the superior performance of our material over the kind of aluminum alloys previously used in the art. The comparative data for our specialized high purity aluminum alloy is with respect to 6061 aluminum alloy, since this is the alloy which has been most commonly used in the industry. We are attaching one pamphlet which is entitled "Innovative Alloy for Chamber Components", which shows the kinds of process conditions the semiconductor apparatus may be exposed to. This pamphlet also shows the particle counts (particles found on surfaces of semiconductor device coupons processed in the apparatus), as a function of the process conditions and the composition of the liner in the apparatus process chamber. A portion of the data is for an anodized liner with a 6061 aluminum alloy substrate. This is compared with data for an anodized liner where the aluminum alloy substrate is our specialized high purity aluminum alloy. Clearly, the particle counts are reduced very significantly when the aluminum alloy used to fabricate the anodized liner is our specialized high purity aluminum alloy. We are attaching a second pamphlet which is entitled "HCL Bubble Test Data". This pamphlet shows comparative data for the HCL Bubble Test described in our patent application. This data shows that 6061 aluminum alloy having an anodized coating produced using an Oxalic acid anodization treatment of the kind we described in our patent application fails the bubble test in about 0.1 hour. However, our specialized high purity aluminum alloy with an anodized layer produced using the same anodization treatment fails the bubble test after more than 24 hours. (We show more than one hour on the block diagram because this one hour time period is considered to be an accomplishment in itself.) This is an indication of the significance of the improvement in semiconductor apparatus lifetime which can be achieved using the specialized aluminum alloy which we are claiming.

14. There is no discussion of the corrosion and particulates problem in the Miyashita et al. reference. The Miyashita et al. performance test for an aluminum oxide coated alloy is how rapidly the aluminum oxide coating is etched away. Our test for performance is the length of

time required for penetration of /diffusion through the coating by reactive plasma species. Our concern is the particulates which fall upon the surface of semiconductor devices being processed in the semiconductor apparatus. As previously discussed the number of particulates produced depends on the integrity of the anodized layer, which is affected by the aluminum alloy substrate characteristics. The integrity of the aluminum alloy is affected by the aluminum alloy initial particle distribution and the maximum concentration of particular mobile impurity elements which are present in the aluminum alloy. It is this combination of factors which determines whether defects are formed in the anodized layer produced on the surface of the aluminum alloy substrate and whether these defects continue to grow during the lifetime of the processing apparatus, providing a source for corrosion of the underlying alloy and the production of particulates. The 6061 alloy discussed above is one of the alloys which is recommended for use in fabrication of the electrodes described in the Miyashita et al. reference. Further, the series of other aluminum alloys recommended in the Miyashita et al reference do not meet the requirements of the specialized aluminum alloy which are recited in our Claims.

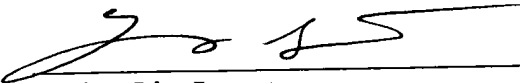
15. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Sec. 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

11/11, 2004

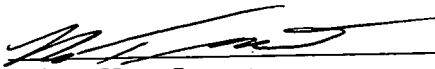
  
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Hong Wang, Inventor



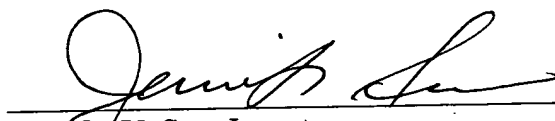
Nov. 10, 2004

  
Yixing Lin, Inventor

Nov 10, 2004

  
Brian West, Inventor

Nov. 11, 2004

  
Jennifer Y. Sun, Inventor

Nov 10, 2004

  
Clifford C. Stow, Inventor

# Innovative Alloy for Chamber Components

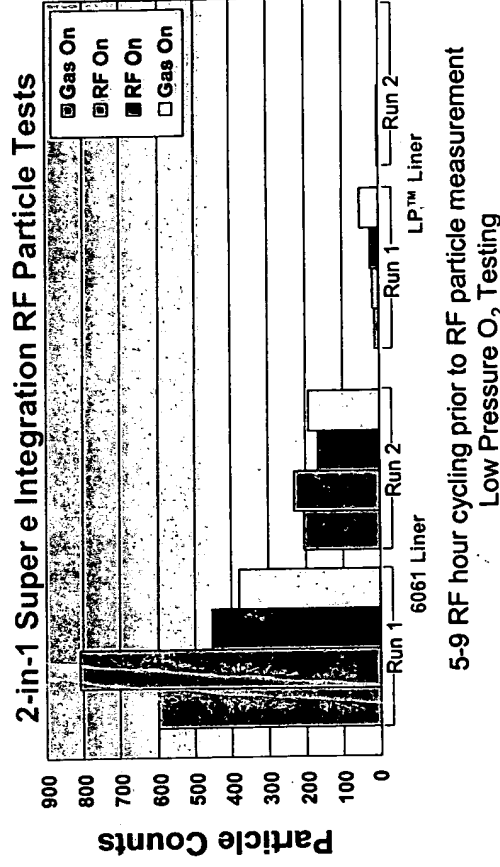
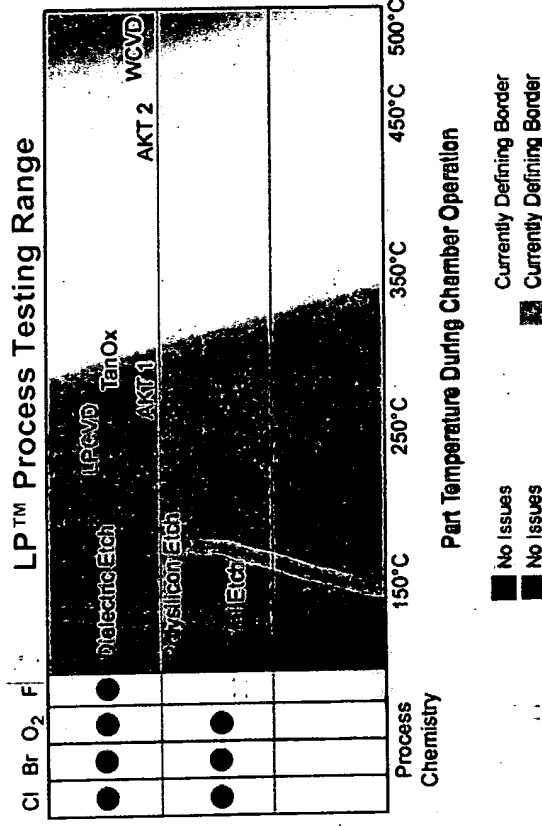
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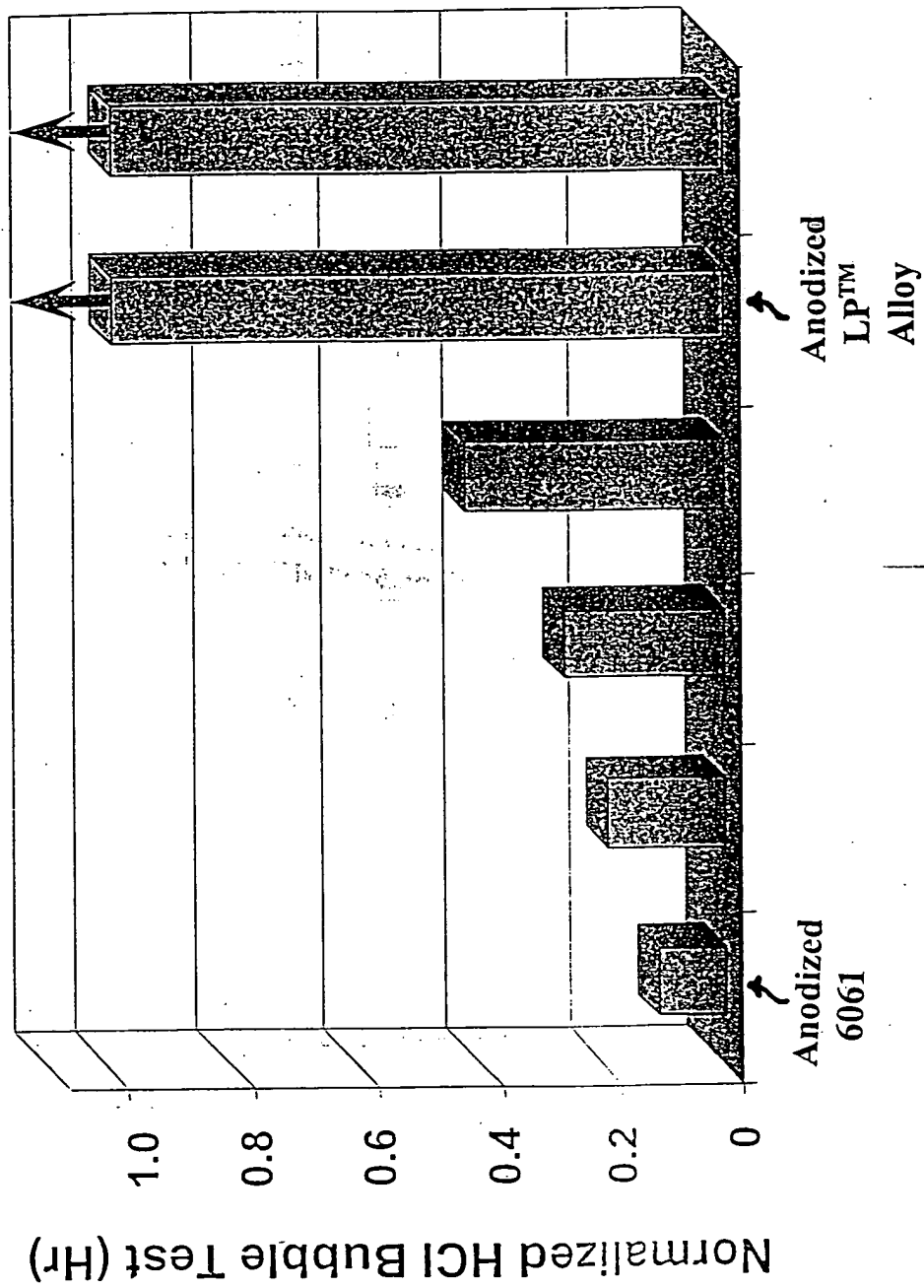
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APPLIED MATERIALS

# HCl Bubble Test Data

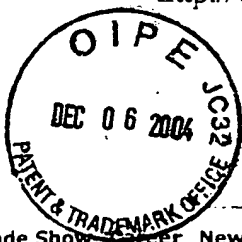


Comparison of HCl Bubble Test Time for LP and 6061 Alloy

80

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engineering fundamentals



Alloys Properties

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## AA 6061

Category Aluminum Alloy

Class Wrought

## Composition

Element	Weight %
Al	97.9
Si	0.60
Cu	0.28
Mg	1.0
Cr	0.20

## Mechanical Properties

Properties		Conditions	
		T (°C)	Treatment
Density ( $\times 1000 \text{ kg/m}^3$ )	2.7	25	
Poisson's Ratio	0.33	25	
Elastic Modulus (GPa)	70-80	25	
Tensile Strength (Mpa)	115		
Yield Strength (Mpa)	48	25	O (Alclad) <a href="#">more</a>
Elongation (%)	25		
Reduction in Area (%)			
Hardness (HB500)	30	25	O <a href="#">more</a>
Shear Strength (MPa)	83	25	O <a href="#">more</a>
Fatigue Strength (MPa)	62	25	O <a href="#">more</a>

## Thermal Properties

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Properties		Conditions	
		T (°C)	Treatment
Thermal Expansion ( $10^{-6}/^{\circ}\text{C}$ )	23.4	20-100	
Thermal Conductivity (W/m-K)	180	25	O <a href="#">more</a>

### Electric Properties

Properties		Conditions	
		T (°C)	Treatment
Electric Resistivity ( $10^{-9}\text{W-m}$ )	37	25	O <a href="#">more</a>

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